

125°, and ash at about 550° until white or nearly white. Cool, add 25 ml (1 + 4) HNO₃, heat on steam bath for ca 30 min., and filter into 400 ml beaker, washing dish and paper thoroly with H₂O. Dil. filtrate to ca 100 ml and proceed as in the quinoline molybdate method for fertilizer which utilizes the quimociac reagent.”¹

(6) That the method for chlorinated hydrocarbon pesticide residues in meat, 23.045, be changed to read: “Prep. sample weighing

from 100–200 g as in 23.001(a), (b), or (c). Dry appropriate portions by evap on steam bath 2 or 3 times with alcohol (200–400 ml). Ext. fat from dried product 3 times with petroleum ether (100–200 ml total), combine exts, and evap. on steam bath. Proceed as in Chapter 24.

“Fatty tissue: Pass chilled sample through food chopper once and render fat in air oven until dry. Det. residue on rendered fat. See Chapter 24.”¹

Report on Microchemical Methods

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The Carius combustion method for chlorine, bromine, and iodine is a reliable official procedure; however, most microanalytical laboratories now use the newer, more rapid oxygen flask combustion procedure for determining these elements. Consequently, a collaborative study of the oxygen flask method was conducted to evaluate this procedure. In this study the collaborators were asked to analyze three samples, one each for chlorine, bromine, and iodine, by the oxygen flask combustion procedure they normally use in their laboratories. Details of each analyst's procedure were obtained and the results were analyzed statistically to try to evaluate the effect of different steps in the methods used by the various collaborators. From this study it is planned to construct a procedure, to be tested next year, which will include those steps of the different methods that statistically are shown to produce the best results as well as those steps which are most commonly used, provided these appear to yield satisfactory analyses.

The results from the preliminary study are very encouraging and should lead to a

reliable oxygen flask combustion procedure for the three halogens.

The method for oxygen was adopted as a first action procedure in 1962 after thorough study by the Associate Referee. Because it has been in use for two years with no adverse criticisms or questions concerning it coming to the Referee's attention and because the results in both the preliminary and final collaborative studies were so good, the method for oxygen is being recommended for adoption as official, final action.

Recommendations

It is recommended—

(1) That study of the oxygen flask combustion method for chlorine, bromine, and iodine be continued.

(2) That the first action procedure for oxygen be adopted as official, final action.

(3) That a study of the oxygen flask combustion method for sulfur be initiated.

(4) That study of methods for molecular weight be continued.

(5) That study of the first action procedure for fluorine be continued.